

HIGH CONCENTRATION METHOD  
STANDARD OPERATING PROCEDURE

Date: January, 1993  
Revision: 0

YES NO N/A

PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER: \_\_\_\_\_ LAB: \_\_\_\_\_

SITE: \_\_\_\_\_

1.0 Data Completeness and Deliverables

1.1 Have any missing deliverables been received  
and added to the data package? ☐ \_\_\_\_ \_\_\_\_

ACTION: Call lab for explanation/resubmittal of any  
missing deliverables. If lab cannot provide  
them, note the effect on the review of the data  
under the "Contract Problems/Non-Compliance  
section of the data assessment.

1.2 Was SMO CCS checklist included with the package? ☐ \_\_\_\_ \_\_\_\_

2.0 Cover Letter, SDG Narrative

2.1 Is the Narrative or Cover Letter Present? ☐ \_\_\_\_ \_\_\_\_

2.2 Are case number and/or SAS number contained  
in the narrative or cover letter? ☐ \_\_\_\_ \_\_\_\_

3.0 Data Validation Checklist

The following High Concentration Checklist is divided  
into three parts. Part one is filled out if the package  
contains any VOA analyses, Part two for any Semivolatile  
analyses, Part three for any Aroclors analyses.

3.1 Does this package contain:

VOA data? \_\_\_\_ \_\_\_\_

Extractables data? \_\_\_\_ \_\_\_\_

Aroclor/Toxaphene data? \_\_\_\_ \_\_\_\_

Action: Complete the corresponding parts of the checklist.

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PART A: VOA ANALYSES

1.0 Traffic Reports and Laboratory Narrative

1.1 Are the Traffic Report Forms present for all samples? ☐ ☐ ☐

ACTION: If no, contact lab for replacement of missing or illegible copies.

1.2 Do the Traffic Reports or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data? ☐ ☐ ☐

ACTION: 1. If any sample analyzed as a soil contains 50% to 90% water, all data should be flagged as estimated, "J." If a soil sample contains more than 90% water, all data should be qualified as unusable, "R."

2. If samples were not iced upon receipt at the laboratory, flag all positive results "J" and all non-Detects "UJ."

3. If both VOA vials for a sample have air bubbles or the VOA vial analyzed had air bubbles, flag all positive results "J" and all non-detects "R."

2.0 Holding Times

2.1 Have any VOA technical holding times, determined from date of collection to date of analysis, been exceeded? ☐ ☐ ☐

Aqueous samples maintained at 4 C must be analyzed within 14 days of validated time of sample receipt (VTSR).

Soils or solid samples must be analyzed within 10 days of VTSR.

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ACTION: If holding times are exceeded, flag all positive results as estimated, "J" and sample quantitation limits as estimated, "UJ," and document in the narrative that holding times were exceeded. If analyses were done more than 14 days beyond holding time, either on the first analysis or upon re-analysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. At a minimum, all results must be qualified "J," but the reviewer may determine that non-detect data are unusable, "R." If holding times are exceeded by more than 28 days, all non detect data are unusable, "R."

3.0 Volatile Surrogate Recovery (Form II HCV)

3.1 Are the Volatile Surrogate Recovery Summaries (Form II HCV) present and complete, with all VOA samples listed on the proper summary form, for each of the following matrices:

a. Water Miscible Liquids (WML)?	<input type="checkbox"/>	___	___
b. Water Immiscible Liquids (WIL)?	<input type="checkbox"/>	___	___
c. Solids?	<input type="checkbox"/>	___	___

ACTION: Call lab for explanation/ resubmittals. If missing deliverables are unavailable, document the effect in the data assessments.

3.2 Were outliers marked correctly with an asterisk? ☐ \_\_\_ \_\_\_

ACTION: Circle all outliers in red pencil.

3.3 Were one or more VOA surrogate compound recoveries outside of contract specifications for any sample, QC sample, or method blank?	___	<input type="checkbox"/>	___
If yes, were samples re-analyzed?	<input type="checkbox"/>	___	___
Were method blanks re-analyzed?	<input type="checkbox"/>	___	___

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ACTION: If recoveries are > 10% but 1 or more compounds fail to meet SOW specifications:

1. Qualify all positive results as estimated, "J."
2. Qualify all non-detects as estimated detection limits, "UJ," where the recovery is less than the lower acceptance limit.
3. If surrogate recoveries are above the upper acceptance limits, do not qualify non-detects.

If any system monitoring compound recovery is < 10%:

1. Flag all positive results as estimated, "J."
2. Flag all non-detects as unusable, "R."

Professional judgement should be used to qualify data that only have method blank surrogate recoveries out of specification in both original and re-analyses. Check the internal standard areas.

3.4 Are there any transcription/calculation errors between raw data and Form II?

\_\_\_ ☐ \_\_\_

ACTION: If large errors exist, call lab for explanation/resubmittal. Make any necessary corrections and note errors in the data assessment.

4.0 Volatile Control Matrix Spike Recovery (Form III HCV)

4.1 Is the Volatile Control Matrix Spike Recovery Form (Form III HCV) present?

☐ \_\_\_ \_\_\_

4.2 Were matrix spikes analyzed at the required frequency for each of the following matrices:

a. Water Miscible Liquid (WML)?

☐ \_\_\_ \_\_\_

b. Water Immiscible Liquid (WIL)?

☐ \_\_\_ \_\_\_

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YES    NO    N/A

c.     Solid ☐    ☐    ☐

ACTION: If any matrix spike data are missing, take the action specified in 3.1 above.

ACTION: Check calculations, surrogates, MS- solutions, and instrument performance.

4.3    How many VOA spike recoveries are outside QC limits?

WML

WIL

Solids

\_\_\_ out of 5

\_\_\_ out of 5

\_\_\_ out of 5

ACTION: No action is taken based on CMS data alone. However, using informed professional judgement, the MS/MSD results may be used in conjunction with other QC criteria to determine the need for qualification of the data.

5.0    Volatile Method Blank (Form IV HCV)

5.1    Is the Method Blank Summary (Form IV HCV) present? ☐    ☐    ☐

5.2    Frequency of Analysis: for the analysis of VOA TCL compounds, has a reagent/method blank been analyzed for each SDG, or every 20 samples of similar matrix (WML, WIL, solid), whichever is more frequent? ☐    ☐    ☐

5.3    Has a VOA method/instrument blank been analyzed at least once every twelve hours for each GC/MS system used? ☐    ☐    ☐

ACTION: If any method blank data are missing, call lab for explanation/ resubmittal. If method blank data are not available, qualify as unusable, "R," all associated positive data. However, using professional judgement, the data reviewer may substitute field blank or trip blank data for missing method blank data.

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YES    NO    N/A

- 5.4 Chromatography: review the blank raw data - chromatograms (RICs), quant reports or data system printouts and spectra.

Is the chromatographic performance (baseline stability) for each instrument acceptable for High Conc. VOAs?

[ ]    —    —

ACTION:    Use professional judgement to determine the effect on the data.

6.0            Contamination

NOTE:        "Water blanks", "drill blanks", and distilled water blanks" are validated like any other sample, and are not used to qualify data. Do not confuse them with the other QC blanks discussed below.

- 6.1 Do any method/instrument/reagent blanks have positive results (TCL and/or TIC) for VOAs? When applied as described below, the contaminant concentration in these blanks are corrected for the sample conversion factor (which includes the sample dilution factor, if any).

—    [ ]    —

- 6.2 Do any field/trip/rinse blanks have positive VOA results (TCL and/or TICs)?

—    [ ]    —

ACTION:    Prepare a list of the samples associated with each of the contaminated blanks.

NOTE:        All field blank results associated to a particular group of samples (may exceed one per case) must be used to qualify data. Trip blanks are used to qualify only those samples with which they were shipped and are not required for solid matrices. Blanks may not be qualified because of contamination in another blank. Field Blanks & Trip Blanks must be qualified for system monitoring compound, instrument performance criteria, spectral or calibration QC problems.

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ACTION: Follow the directions in the table below to qualify TCL results due to contamination. Use the largest value from all the associated blanks. If any of the blanks are grossly contaminated, all associated sample data should be qualified as unusable ("R").

Table 5:

BLANK CONTAMINATION	Sample conc < CRQL and < 10x blank result	Sample conc > CRQL but < 10x blank result	Sample conc > CRQL and > 10x blank result
Methylene chloride acetone toluene	Report CRQL and qualify "U"	Flag sample result with a "U"	No qualification is necessary

	Sample conc < CRQL and < 5x blank result	Sample conc > CRQL but < 5x blank result	Sample conc > CRQL and > 5x blank result
Other contaminants	Report CRQL and qualify "U"	Flag sample result with a "U"	No qualification is necessary

NOTE: Analytes qualified "U" for blank contamination are still considered as "hits" when qualifying for calibration criteria.

ACTION: For TIC compounds, if the concentration in the sample is less than five times the concentration in the most contaminated associated blank, flag the sample data "R," unusable.

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6.3 Are there field/rinse/equipment blanks associated with every sample?

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ACTION: Note in data assessment that there is no associated field/rinse/equipment blank.

Exception: samples taken from a drinking water tap do not have associated field blanks.

7.0 GC/MS Instrument Performance Check (Form V HCV)

7.1 Are the GC/MS Instrument Performance Check Forms (Form V HCV) present for Bromofluorobenzene (BFB)?

☐ ☐ ☐

7.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the BFB provided for each 12 hour shift?

☐ ☐ ☐

7.3 Has an instrument performance compound been analyzed for every twelve hours of sample analysis per instrument?

☐ ☐ ☐

ACTION: List the date, time, instrument ID, and sample number(s) for which no associated GC/MS tuning data are available.

DATE	TIME	INSTRUMENT	SAMPLE NUMBER(S)
------	------	------------	------------------

_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

ACTION: If lab cannot provide missing data, reject (qualify "R") all data generated outside an acceptable twelve hour calibration interval.

7.4 Have the ion abundances been normalized to m/z 95?

☐ ☐ ☐



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ACTION: If mass assignment is in error, qualify all associated data as unusable ("R").

7.5 Have the ion abundance criteria been met for each instrument used? [ ]

ACTION: List all data which do not meet ion abundance criteria (attach a separate sheet).

ACTION: If ion abundance criteria are not met, the Region II TPO must be notified.

7.6 Are there any transcription/calculation errors between mass lists and Form Vs? (Check at least two values but if errors are found, check more.) [ ]

7.7 Have the appropriate number of significant figures (two) been reported? [ ]

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and document the effect in the data assessments.

7.8 Are the spectra of the mass calibration compound acceptable? [ ]

ACTION: Use professional judgement to determine whether associated data should be accepted, qualified, or rejected.

8.1 Are the Organic Analysis Data Sheets (Form I HCV), VOA chromatograms, mass spectra for the identified compounds and the data system printouts (quant reports) included in the sample package for each of the following:

a. Samples and/or fractions as appropriate? ☐ ☐ ☐

b. Matrix spikes? [ ]

c.     Blanks?                        [ ] \_\_\_\_\_

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	YES	NO	N/A
ACTION: If any data are missing, take action specified in 3.2 above.			
8.2 Are the response factors shown in the quant report?	<input type="checkbox"/>	___	___
8.3 Is chromatographic performance acceptable with respect to:			
Baseline stability?	<input type="checkbox"/>	___	___
Resolution?	<input type="checkbox"/>	___	___
Peak shape?	<input type="checkbox"/>	___	___
Full-scale graph (attenuation)?	<input type="checkbox"/>	___	___
Other: _____?	<input type="checkbox"/>	___	___
ACTION: Use professional judgement to determine the acceptability of the data.			
8.4 Are the lab-generated standard mass spectra of the identified VOA compounds present for each sample?	<input type="checkbox"/>	___	___
ACTION: If any mass spectra are missing, take action specified in 3.2 above. If lab does not generate their own standard spectra, make note in "Contract Problems/Non-compliance".			
8.5 Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration?	<input type="checkbox"/>	___	___
8.6 Are all ions present in the standard mass spectrum at a relative intensity greater than 10% also present in the sample mass spectrum?	<input type="checkbox"/>	___	___
8.7 Do sample and standard relative ion intensities agree within 20%?	<input type="checkbox"/>	___	___
ACTION: Use professional judgement to determine			

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acceptability of data. If it is determined that incorrect identifications were made, all such data should be rejected (R), flagged "N" (presumptive evidence of the presence of the compound) or changed to not detected (U) at the calculated detection limit. In order to be positively identified, the data must comply with the criteria listed in sections 8.5, 8.6, and 8.7 above.

## 9.0 Tentatively Identified Compounds (Form I HCV-TIC)

9.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:

b. Blanks? [ ]

ACTION: Add "JN" qualifier if missing.

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9.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% also present in the sample mass spectrum? ☐ ☐ ☐

9.5 Do TIC and "best match" standard relative ion intensities agree within 20%? ☐ ☐ ☐

ACTION: Use professional judgement to determine acceptability of TIC identifications. If it is determined that an incorrect identification was made, change identification to "unknown" or to some less specific identification (example: "substituted benzene") as appropriate.

Also, when a compound is not found in any blank, but is detected in a sample and is a suspected artifact of a common laboratory contaminant, the result should be qualified as unusable ("R"). Some common laboratory contaminants include CO<sub>2</sub> (m/z = 44), siloxanes m/z = 73), hexane, aldol condensation products of acetone, solvent preservatives (such as cyclohexene and related by-products: cyclohexanone, cyclohexanol, chlorocyclohexene, etc.) and certain freons.

### 10.0 Compound Quantitation and Reported Detection Limits

10.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values. Verify that the correct internal standard, quantitation ion, and RRF were used to calculate Form I results. Were any errors found? ☐ ☐ ☐

10.2 Are the CRQLs adjusted to reflect sample dilutions (check the "Conversion Factors" on Form Is for accuracy)? ☐ ☐ ☐

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and note errors under "Conclusions".

ACTION: When a sample is analyzed at more than one dilution, the lowest CRQLs are used (unless a

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QC problem dictates the use of the higher CRQL data from the diluted sample analysis). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and its associated value on the original Form I and substituting the data from the analysis of the diluted sample. Specify which Form I is to be used, then draw a red "X" across the entire page of all Form Is that should not be used, including any in the summary package.

11.0 GC/MS Initial Calibration (Form VI)

11.1 Are the Initial Calibration Forms (Form VI HCV), chromatograms and data system printouts (quant reports) present and complete for the volatile initial calibration standards at concentrations of 20, 50, 100, 150 and 200 ug/l?    ☐    ☐    ☐

ACTION:    If any initial calibration data are missing, take action specified in 3.1 above.

11.2 Are all the response factors stable for VOA's over the entire concentration range of the calibration (% Relative Standard Deviation (%RSD)  $\leq$  30.0%)?    ☐    ☐    ☐

ACTION:    Circle all outliers in red.

NOTE:    Although 11 VOA compounds have a minimum RRF and maximum %RSD as specified on Form VI HCV, the technical criteria are the same for all analytes.

ACTION:    If %RSD > 30.0%, qualify associated positive results for that analyte "J" and non-detects using professional judgement. When RSD > 90%, flag all non-detects for that analyte "R," unusable.

NOTE:    Analytes previously qualified "U" for blank contamination are still considered as "hits" when qualifying for initial calibration criteria.

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11.3 Are the RRFs above 0.05 for all TCL analytes? ☒ ☐ ☐

Action: Circle all outliers in red.

Action: If any RRF are < 0.05, qualify associated non-detects as unusable, "R," and flag associated positive data as estimated, "J."

11.4 Are there any transcription/calculation errors in the reported relative response factors (RRF), average response factors ( $\overline{RRF}$ ), or %RSD? (Calculate at least 2 values using raw data. If errors are found, check more.) ☐ ☒ ☐

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ACTION: Circle errors in red.

ACTION: If errors are large, call the lab for explanation/resubmittal. Make any necessary corrections and note errors under "Conclusions".

12.0 GC/MS Continuing Calibration (Form VII HCV)

12.1 Are the Continuing Calibration Forms (Form VII HCV) present and complete for the VOA fraction?

☐ ☐ ☐

12.2 Has a continuing calibration standard been analyzed for every twelve hours of period of sample analysis per instrument?

☐ ☐ ☐

ACTION: List below all samples that were not analyzed within twelve hours of the previous continuing calibration standard.

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ACTION: If any forms are missing or no continuing calibration standard has been analyzed within twelve hours of every sample analysis, call lab for explanation/resubmittal. If continuing calibration data are not available, flag all associated sample data as unusable, "R."

12.3 Is the % Difference (%D) between the initial and continuing RRF > 25% for any VOA TCL analyte?

☐ ☐ ☐

ACTION: Circle all outliers in red.

ACTION: Qualify both positive results and non-detects for the outlier compound(s) as estimated. When %D is above 90%, qualify all non-detects for that analyte as unusable, "R."

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12.4 Is the RRF < 0.05 for any volatile compounds? ☐ ☐ ☐

ACTION: Circle all outliers in red.

ACTION: If the RRF < 0.05, qualify associated non-detects as unusable, "R," and associated positive results as estimated, "J."

12.5 Are there any transcription/calculation errors in the reported average response factors ( $\overline{RRF}$ ), relative response factors (RRF), or % difference (%D) between initial and continuing RRFs? (Calculate at least two values from raw data; If errors are found, check more.) ☐ ☐ ☐

ACTION: Circle errors in red.

ACTION: If errors are large, call the lab for explanation/resubmittal. Make any necessary corrections and note errors under "Conclusions".

13.0 Internal Standard (Form VIII HCV)

13.1 Are the internal standard areas (Form VIII HCV) of every sample and blank within the upper and lower limits (-50% to + 100%) for each continuing calibration? ☐ ☐ ☐

ACTION: List all the outliers below.

Sample#	Internal Std	Area	Lower Limit	Upper Limit
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____



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(Attach additional sheets if necessary.)

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- ACTION:
1. If the internal standard area count is outside the upper or lower limit, flag all positive results quantitated with this internal standard with a "J."
  2. Non-detects associated with IS area counts > 100% should not be qualified.
  3. If IS area is below the lower limit (< 50%), qualify all associated non-detects (U values) "J". If extremely low area counts are reported, (< 25%), or if performance exhibits a major abrupt drop off, flag all associated non-detects as unusable ("R").

13.2 Are the internal standard retention times in each sample within 30 seconds of the corresponding retention times in the associated calibration standard?

☐ ☐ ☐

ACTION: Professional judgement should be used to qualify sample data if the internal standard retention times differ by more than 30 seconds.

14.0 Field Duplicates

15.1 Were any field duplicates submitted for High Conc. VOA analysis?

☐ ☐ ☐

ACTION: Compare the field duplicates and calculate the relative percent difference between the corresponding positive results.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative. However, if large differences exist, confirm the identification of the field duplicates by contacting the sampler.

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PART B: HIGH CONCENTRATION EXTRACTABLE ANALYSES

1.0        Traffic Reports and Laboratory Narrative

1.1    Are the Traffic Report Forms present for all samples?        ☐    ☐    ☐

ACTION:    If no, contact lab for replacement of missing or illegible copies.

1.2    Do the Traffic Reports or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data?        ☐    ☐    ☐

ACTION:    If samples were not iced upon receipt at the laboratory, flag all positive results "J" and all non-Detects "UJ."

NOTE:        If any samples were multiphasic, containing a separate phase which is at least 10% of the amount of the main sample, the phases should have been separated and analyzed as individual subsamples.

1.3        Does the laboratory narrative or extraction log contain a record of sample pH determinations?        ☐    ☐    ☐

ACTION:    If not, contact the lab for explanation/resubmittals. If the data is unavailable, or sample pH was not properly adjusted prior to extraction, use professional judgement to determine the effect on analytical results and document this in the data assessment.

2.0        Holding Times

2.1    Have any Extractables holding times been exceeded, determined from validated time of sample receipt (VTSR) to date of analysis ?        ☐    ☐    ☐

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YES    NO    N/A

ACTION: The contract requires extracts to be analyzed within 40 days of VTSR. If holding times are exceeded, flag all positive results as estimated, "J" and sample quantitation limits as estimated, "UJ", and document in the narrative that holding times were exceeded. If analyses were done more than 14 days beyond holding time, either on the first analysis or upon re-analysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. At a minimum, all results must be qualified "J," but the reviewer may determine that non-detect data are unusable, "R." If holding times are exceeded by more than 28 days, all non detect data are unusable, "R."

3.0 Extractable Surrogate Recovery (Form II HCE)

3.1 Are the Extractable Surrogate Recovery Summaries (Form II HCE) present and complete, with all Extractables samples listed on the proper summary form, for each of the following matrices:

- |                                    |                          |     |     |
|------------------------------------|--------------------------|-----|-----|
| a. Water Miscible Liquids (WML)?   | <input type="checkbox"/> | ___ | ___ |
| b. Water Immiscible Liquids (WIL)? | <input type="checkbox"/> | ___ | ___ |
| c. Solids?                         | <input type="checkbox"/> | ___ | ___ |

ACTION: Call lab for explanation/ resubmittals. If missing deliverables are unavailable, document the effect in the data assessments.

3.2 Were outliers marked correctly with an asterisk? ☐ \_\_\_ \_\_\_

ACTION: Circle all outliers in red pencil.

3.3 Were two or more Extractables surrogate compound recoveries outside of contract specifications for any sample, QC sample, or method blank? \_\_\_ ☐ \_\_\_

If yes, were samples re-analyzed? ☐ \_\_\_ \_\_\_

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Were method blanks re-analyzed?

☐ ☐ ☐

ACTION: If all extractable surrogate recoveries are > 10% but two within the base-neutral or acid fraction do not meet SOW specifications, for the affected fraction only (i.e. base-neutral or acid compounds) :

1. Qualify all positive results as estimated, "J."
2. Qualify all non-detects as estimated detection limits, "UJ," where the recovery is less than the lower acceptance limit.
3. If surrogate recoveries are above the upper acceptance limits, do not qualify non-detects.

3.4 If any system monitoring compound recovery is < 10%:

1. Flag all positive results for the affected fraction as estimated, "J."
2. Flag all non-detects as unusable, "R."

Professional judgement should be used to qualify data that only have method blank surrogate recoveries out of specification in both original and re-analyses. Check the internal standard areas.

3.5 Are there any transcription/calculation errors between raw data and Form II?

☐ ☐ ☐

ACTION: If large errors exist, call lab for explanation/resubmittal. Make any necessary corrections and note errors in the data assessment.

4.0 Extractable Control Matrix Spike Recovery (Form III HCE)

4.1 Is the Extractable Control Matrix Spike Recovery Form (Form III HCE) present?

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YES    NO    N/A

4.2 Were matrix spikes analyzed at the required frequency for each of the following matrices:

a. Water Miscible Liquid (WML)?	<input type="checkbox"/>	___	___
b. Water Immiscible Liquid (WIL)?	<input type="checkbox"/>	___	___
c. Solid ?	<input type="checkbox"/>	___	___

ACTION: If any matrix spike data are missing, take the action specified in 3.1 above.

ACTION: Check calculations, surrogates, MS solutions, and instrument performance.

4.3 How many Extractables spike recoveries are outside QC limits?

<u>WML</u>	<u>WIL</u>	<u>Solids</u>
___ out of 13	___ out of 13	___ out of 13

ACTION: No action is taken based on CMS data alone. However, using informed professional judgement, the CMS results may be used in conjunction with other QC criteria to determine the need for qualification of the data.

5.0 Extractable Method Blank (Form IV HCE)

5.1 Is the Method Blank Summary (Form IV HCE) present?	<input type="checkbox"/>	___	___
5.2 Frequency of Analysis: for the analysis of Extractables TCL compounds, has a reagent/method blank been analyzed for each SDG, or every 20 samples of similar matrix whichever is more frequent?	<input type="checkbox"/>	___	___
5.3 Has an Extractables method/instrument blank been analyzed at least once every twelve hours for each GC/MS system used?	<input type="checkbox"/>	___	___

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YES   NO   N/A

ACTION:    If any method blank data are missing, call lab for explanation/ resubmittal. If method blank data are not available, qualify as unusable, "R," all associated positive data. However, using professional judgement, the data reviewer may substitute field or rinse blank data for missing method blank data.

5.4   Chromatography: review the blank raw data - chromatograms (RICs), quant reports or data system printouts and spectra.

Is the chromatographic performance (baseline stability) for each instrument acceptable for High Conc. Extractables?

[ ]    \_\_\_\_\_

\_\_\_\_\_

ACTION:    Use professional judgement to determine the effect on the data.

6.0        Contamination

NOTE:        "Water blanks", "drill blanks", and distilled water blanks" are validated like any other sample, and are not used to qualify data. Do not confuse them with the other QC blanks discussed below.

6.1   Do any method/instrument/reagent blanks have positive results (TCL and/or TIC) for Extractables? When applied as described below, the contaminant concentration in these blanks are corrected for the sample conversion factor (which includes the sample dilution factor, if any).

\_\_\_\_\_ [ ] \_\_\_\_\_

6.2   Do any field/trip/rinse blanks have positive Extractables results (TCL and/or TICs)?

\_\_\_\_\_ [ ]

\_\_\_\_\_

ACTION:    Prepare a list of the samples associated with each of the contaminated blanks.

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YES NO N/A

NOTE: All field blank results associated with a particular group of samples (may exceed one per case) must be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field Blanks must be qualified for system monitoring compound, instrument performance criteria, spectral or calibration QC problems.

ACTION: Follow the directions in the table below to qualify TCL results due to contamination. Use the largest value from all the associated blanks. If any of the blanks are grossly contaminated, all associated sample data should be qualified as unusable.

BLANK CONTAMINATION	Sample conc < CRQL and < 10x blank result	Sample conc > CRQL but < 10x blank result	Sample conc > CRQL and > 10x blank result
Phthalates	Report CRQL and qualify "U"	Flag sample result with a "U"	No qualification is necessary

	Sample conc < CRQL and < 5x blank result	Sample conc > CRQL but < 5x blank result	Sample conc > CRQL and > 5x blank result
Other contaminants	Report CRQL and qualify "U"	Flag sample result with a "U"	No qualification is necessary

NOTE: Analytes qualified "U" for blank contamination are still considered as "hits" when qualifying for calibration criteria.

ACTION: For TIC compounds, if the concentration in the sample is less than five times the concentration in the most contaminated



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associated blank, flag the sample data "R"  
(unusable).

6.3 Are there field/rinse/equipment blanks associated with every sample? [ ]    \_\_\_    \_\_\_

ACTION: Note in data assessment that there is no associated field/rinse/equipment blank.

Exception: samples taken from a drinking water tap do not have associated field blanks.

7.0 GC/MS Tuning and Mass Calibration (Form V HCE)

7.1 Are the GC/MS Tuning and Mass Calibration Forms (Form V HCE) present for Decafluorotriphenylphosphine (DFTPP)? [ ]    \_\_\_    \_\_\_

7.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the DFTPP provided for each 12 hour shift? [ ]    \_\_\_    \_\_\_

7.3 Has an instrument performance compound been analyzed for every twelve hours of sample analysis per instrument? [ ]    \_\_\_    \_\_\_

ACTION: List the date, time, instrument ID, and sample number(s) for which no associated GC/MS tuning data are available.

DATE	TIME	INSTRUMENT	SAMPLE NUMBER(S)
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

ACTION: If lab cannot provide missing data, reject (qualify "R") all data generated outside an acceptable twelve hour calibration interval.

7.4 Have the ion abundances been normalized to m/z 198 ? [ ]    \_\_\_    \_\_\_

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ACTION: If mass assignment is in error, qualify all associated data as "R", (unusable).

7.5 Have the ion abundance criteria been met for each instrument used? [ ]

ACTION: List all data which do not meet ion abundance criteria (attach a separate sheet).

ACTION: If ion abundance criteria are not met, the Region II TPO must be notified.

7.6 Are there any transcription/calculation errors between mass lists and Form Vs? (Check at least two values but if errors are found, check more.) [ ]

7.7 Have the appropriate number of significant figures (two) been reported? [ ]

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and document the effect in the data assessments.

7.8 Are the spectra of the mass calibration compound acceptable? [ ]

ACTION: Use professional judgement to determine whether associated data should be accepted, qualified, or rejected.

8.1 Are the Organic Analysis Data Sheets (Form I HCE), Extractables chromatograms, mass spectra for the identified compounds and the data system printouts (quant reports) included in the sample package for each of the following:

a. Samples and/or fractions as appropriate? ☐ ☐ ☐

b. Matrix spikes? [ ]

c. Blanks? [ ]

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YES NO N/A

ACTION: If any data are missing, take action specified in 3.1 above.

8.2 Are the Conversion factors entered in the Form I's?

☐ ☐ ☐

NOTE: Check the calculation of the Conversion Factor for at least two samples.

8.3 Are the response factors shown in the quant report?

☐ ☐ ☐

8.4 Has GPC cleanup been performed on all sample extracts?

☐ ☐ ☐

ACTION: If data suggests that GPC was not performed, use professional judgement. Make note in "Contract Problems/Non-compliance".

8.5 Is chromatographic performance acceptable with respect to:

Baseline stability?

☐ ☐ ☐

Resolution?

☐ ☐ ☐

Peak shape?

☐ ☐ ☐

Full-scale graph (attenuation)?

☐ ☐ ☐

Other: \_\_\_\_\_?

☐ ☐ ☐

ACTION: Use professional judgement to determine the acceptability of the data.

8.6 Are the lab-generated standard mass spectra of the identified Extractables compounds present for each sample?

☐ ☐ ☐

ACTION: If any mass spectra are missing, take action specified in 3.1 above. If lab does not generate their own standard spectra, make note in "Contract Problems/Non-compliance". If spectra are missing reject all positive data.

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YES NO N/A

- 8.7 Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration? [ ]    \_\_\_    \_\_\_
- 8.8 Are all ions present in the standard mass spectrum at a relative intensity greater than 10% also present in the sample mass spectrum? [ ]    \_\_\_    \_\_\_
- 8.9 Do sample and standard relative ion intensities agree within 20%? [ ]    \_\_\_    \_\_\_

ACTION: Use professional judgement to determine acceptability of data. If it is determined that incorrect identifications were made, all such data should be rejected (R), flagged "N" (presumptive evidence of the presence of the compound) or changed to not detected (U) at the calculated detection limit. In order to be positively identified, the data must comply with the criteria listed in sections 8.7, 8.8, and 8.9 above.

ACTION: When sample carry-over is a possibility, professional judgement should be used to determine if instrument cross-contamination has affected any positive compound identification.

9.0 Tentatively Identified Compounds (Form I HCE-TIC)

- 9.1 Are all Tentatively Identified Compound Forms (Form I-HCE) present; and do listed TICs include scan number or retention time, estimated concentration and "J" qualifier? [ ]    \_\_\_    \_\_\_

NOTE: Add the "N" qualifier to all TICs which are identified by a CAS No.

- 9.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:

- a. Samples and/or fractions as appropriate? [ ]    \_\_\_    \_\_\_

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YES NO N/A

[ ]      \_\_\_\_\_

[ ]

[ ]

[ ]

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YES NO N/A

quantitation ion, and RRF were used to calculate  
Form I results. Were any errors found?

\_\_\_ ☐ \_\_\_

10.2 Are the CRQLs adjusted to reflect sample dilutions  
(check the "Conversion Factors" on Form Is for  
accuracy)?

☐ \_\_\_ \_\_\_

ACTION: If errors are large, call lab for  
explanation/resubmittal, make any necessary  
corrections and note errors under  
"Conclusions".

ACTION: When a sample is analyzed at more than one  
dilution, the lowest CRQLs are used (unless a  
QC problem dictates the use of the higher  
CRQL data from the diluted sample analysis).  
Replace concentrations that exceed the  
calibration range in the original analysis by  
crossing out the "E" and its associated value  
on the original Form I and substituting the  
data from the analysis of the diluted sample.  
Specify which Form I is to be used, then draw  
a red "X" across the entire page of  
all Form Is that should not be used, including  
any in the summary package.

11.0 GC/MS Initial Calibration (Form VI HCE)

11.1 Are the Initial Calibration Forms (Form VI HCE),  
chromatograms and data system printouts (quant  
reports) present and complete for the extractable  
initial calibration standards at concentrations  
of 50, 80, 160 mg/l?

☐ \_\_\_ \_\_\_

ACTION: If any initial calibration data are missing,  
take action specified in 3.1 above.

11.2 Are all the response factors stable for Extractables's  
over the entire concentration range of the  
calibration (% Relative Standard Deviation  
(%RSD)  $\leq$  30.0%)?

☐ \_\_\_ \_\_\_

ACTION: Circle all outliers in red.

NOTE: Although 11 Extractables compounds have a

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minimum RRF and maximum %RSD as specified on Form VI HCV, the technical criteria are the same for all analytes.

ACTION:    If %RSD > 30.0%, qualify associated positive results for that analyte "J" and non-detects using professional judgement. When RSD > 90%, flag all non-detects for that analyte "R," unusable.

NOTE:       Analytes previously qualified "U" for blank contamination are still considered as "hits" when qualifying for initial calibration criteria.

11.3 Are the RRFs above 0.05 for all TCL analytes?    ☐    \_\_\_    \_\_\_

Action:    Circle all outliers in red.

Action:    If any RRF are < 0.05, qualify associated non-detects as unusable, "R," and flag associated positive data as estimated, "J."

11.4 Are there any transcription/calculation errors in the reported relative response factors (RRF), average response factors (RRF), or %RSD? (Calculate at least 2 values using raw data. If errors are found, check more.)    \_\_\_    ☐    \_\_\_

ACTION:    Circle errors in red.

ACTION:    If errors are large, call the lab for explanation/resubmittal. Make any necessary corrections and note errors under "Conclusions".

12.0 GC/MS Continuing Calibration (Form VII HCE)

12.1 Are the Continuing Calibration Forms (Form VII HCE) present and complete for the Extractables fractions?    ☐    \_\_\_    \_\_\_

12.2 Has a continuing calibration standard been analyzed for every twelve hour period of sample analysis per instrument?    ☐    \_\_\_    \_\_\_

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YES   NO   N/A

ACTION:   List below all samples that were not  
             analyzed within twelve hours of the  
             previous continuing calibration standard.

---

---

---

ACTION:   If any forms are missing or no continuing  
             calibration standard has been analyzed within  
             twelve hours of every sample analysis, call  
             lab for explanation/resubmittal. If continuing  
             calibration data are not available, flag all  
             associated sample data as unusable, "R."

12.3 Is the % Difference (%D) between the initial average and  
      continuing RRF > 25% for any Extractables TCL  
      analyte? \_\_\_ ☐ \_\_\_

ACTION:   Circle all outliers in red.

ACTION:   Qualify both positive results and non-detects  
             for the outlier compound(s) as estimated.  
             When %D is above 90%, qualify all non-detects  
             for that analyte as unusable, "R."

12.4 Is the RRF < 0.05 for any volatile compounds? ☐ \_\_\_

ACTION:   Circle all outliers in red.

ACTION:   If the RRF < 0.05, qualify associated non-  
             detects as unusable, "R," and associated  
             positive results as estimated, "J."

12.5 Are there any transcription/calculation errors  
      in the reported average response factors (RRF),  
      relative response factors (RRF), or % difference  
      (%D) between initial and continuing RRFs?  
      (Calculate at least two values from raw data;  
      If errors are found, check more.) \_\_\_ ☐ \_\_\_

ACTION:   Circle errors in red.



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YES NO N/A

ACTION: If errors are large, call the lab for explanation/resubmittal. Make any necessary corrections and note errors under "Conclusions".

13.0 Internal Standard (Form VIII HCE)

13.1 Are the internal standard areas (Form VIII HCE) of every sample and blank within the upper and lower limits (-50% to + 100%) for each continuing calibration? [ ] \_ \_

ACTION: List all the outliers below.

Sample#	Internal Std	Area	Lower Limit	Upper Limit
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

(Attach additional sheets if necessary.)

ACTION:

1. If the internal standard area count is outside the upper or lower limit, flag all positive results quantitated with this internal standard with a "J."
2. Non-detects associated with IS area counts > 100% should not be qualified.
3. If IS area is below the lower limit (< 50%), qualify all associated non-detects (U values) "J". If extremely low area counts are reported, (< 25%), or if performance exhibits a major abrupt drop off, flag all associated non-detects as unusable ("R").

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YES NO N/A

13.2 Are the internal standard retention times in each sample within 30 seconds of the corresponding retention times in the associated calibration standard?

☐ ☐ ☐

ACTION: Professional judgement should be used to qualify sample data if the internal standard retention times differ by more than 30 seconds.

14.0 Field Duplicates

15.1 Were any field duplicates submitted for High Conc. Extractables analysis?

☐ ☐ ☐

ACTION: Compare the field duplicates and calculate the relative percent difference between the corresponding positive results.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative. However, if large differences exist, confirm the identification of the field duplicates by contacting the sampler.

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YES    NO    N/A

PART C: AROCLOR/TOXAPHENE ANALYSIS

1.0        Traffic Reports and Laboratory Narrative

- 1.1    Are Traffic Report Forms present for all samples?

[ ]    —    —

ACTION: If no, contact lab for replacement of missing or illegible copies.

- 1.2    Do the Traffic Reports or SDG Narrative indicate any problems with sample receipt, condition of the samples, analytical problems or special circumstances affecting the quality of the data?

—    [ ]    —

ACTION: If samples were not iced upon receipt at the laboratory, flag all positive results "J" and all non-detects "UJ".

- 1.3    High concentration samples are initially separated into individual phases. An aliquot of each phase is transferred to a separate vial and labelled with a unique phase identifier (HC SOW B-23).

2.0        Holding Times

- 2.1    Have any PCB/TOX technical holding times, determined from date of validated time of sample receipt (VTSR), been exceeded?

—    [ ]    —

Sonication extraction of samples for PCB/TOX analysis must be started within 7 days of VTSR.  
Extracts must be analyzed within 40 days of extraction.

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YES NO N/A

ACTION: If technical holding times are exceeded, flag all positive results as estimated (J) and sample quantitation limits (UJ) and document in the narrative that holding times were exceeded.  
If analyses were done more than 14 days beyond holding time, either on the first analysis or upon re-analysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. At a minimum, all the data should at least be qualified "J", but the reviewer may determine that non-detects are unusable (R).

3.0 Surrogate Recovery (Form II HCA)

3.1 Is the Aroclor surrogate recovery form (Form II HCA) present?

☐ ☐ ☐

ACTION: Call lab for explanation/resubmittals.  
If missing deliverables are unavailable, document effect in data assessments.

3.2 Were outliers marked correctly with an asterisk?

☐ ☐ ☐

ACTION: Circle all outliers in red pencil.

3.3 Were surrogate recoveries of TMX or DCB outside of the contract specification for any sample or blank? (40-120%)?

☐ ☐ ☐

ACTION: No qualification is done if surrogates are diluted out. If recovery for both surrogates is below the contract limit, but above 10%, flag all results for that sample 'J'. If recovery is < 10% for either surrogate, qualify positive

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YES NO N/A

results "J" and flag non-detects "R".  
If recovery is above the contract advisory  
limits for both surrogates qualify positive  
values "J".

- 3.4 Were surrogate retention times (RT) within  
the windows established during the initial  
3-point calibration?

☐ ☐ ☐

NOTE: Average RT of surrogate must be  
calculated using all 26 injections  
of initial calibration.

ACTION: If the RT limits are not met, the  
analysis may be qualified unusable (R)  
for that sample on the basis of  
professional judgement.

- 3.5 Are there any transcription/calculation  
errors between raw data and Form II?

☐ ☐ ☐

NOTE: Mean response for each surrogate in  
low point initial calibration analysis  
is used as surrogate CF.

ACTION: If large errors exist, call lab for  
explanation/resubmittal. Make any  
necessary corrections and document  
effect in data assessments.

4.0 AROCLOR CONTROL MATRIX SAMPLE (CMS)

- 4.1 Is the Aroclor Control Matrix Sample (CMS)  
Recovery Form (Form III-HCA) present?

☐ ☐ ☐

- 4.2 Was the CMS analyzed at the required fre-  
quency (once per SDG, or every 20 samples)  
for the High Conc. Aroclor fraction?

☐ ☐ ☐

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YES NO N/A

ACTION: If any CMS data are missing, take the action specified in 3.1 above.

ACTION: Check calculations, surrogates, CMS solutions and instrument performance.

5.0 Blanks (Form IV HCA)

5.1 Is the Method Blank Summary (Form IV HCA) present?

☐ \_\_\_\_

5.2 Frequency of Analysis: For the analysis of Aroclor/Toxaphene compounds, has a method blank been analyzed concurrently for each SDG or every 20 samples or each extraction batch, whichever is more frequent?

☐ \_\_\_\_

ACTION: If any blank data are missing, take the action specified above in 3.1. If blank data is not available, reject (R) all associated positive data.

However, using professional judgement, the data reviewer may substitute field blank data for missing method blank data.

5.3 A separate blank and Form IV should be present if sulfur clean-up was not performed on all of the samples in an extraction batch. Therefore some samples will be listed on two blank summary forms, once under method blank and once under sulfur clean-up blank. Is this additional blank and Form IV present?

☐ \_\_\_\_

ACTION: If sulfur blank data and Form IV are missing, take the action specified in 3.1 above.

5.4 Has a Aroclor instrument blank been analyzed at the beginning and end of every 12 hr. period following the initial calibration sequence

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YES NO N/A

(minimum contract requirement)? ☐ ☐ ☐

- 5.5 A separate instrument blank form must be submitted for each instrument blank analyzed with appropriate samples listed for each blank. Are the additional Form IV-HCA-2 present? ☐ ☐ ☐

ACTION: If any blank data are missing, call lab for explanation/resubmittals. If missing deliverables are unavailable, document the effect in data assessments.

- 5.6 Chromatography: review the blank raw data - chromatograms, quant reports or data system printouts.

Is the chromatographic performance (baseline stability) for each instrument acceptable for Aroclors and Toxaphene? ☐ ☐ ☐

ACTION: Use professional judgement to determine the effect on the data.

6.0 Contamination

NOTE: "Water blanks", "distilled water blanks" and "drilling water blanks" are validated like any other sample and are not used to qualify the data. Do not confuse them with the other QC blanks discussed below.

- 6.1 Do any method/instrument/cleanup blanks have positive results for Aroclors/Toxaphene? When applied as described below, the contaminant concentration in these blanks are multiplied by the sample dilution factor. ☐ ☐ ☐

- 6.2 Do any field/rinse blanks have positive Aroclor/Toxaphene results? ☐ ☐ ☐

ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet)

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YES NO N/A

NOTE: All field blank results associated to a particular group of samples (may exceed one per case or one per day) may be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field blanks must be qualified for surrogate, or calibration QC problems.

ACTION: Follow the directions in the table below to qualify TCL results due to contamination. Use the largest value from all the associated blanks.

Sample conc > CRQL but < 5x blank	Sample conc < CRQL & is < 5x blank value	Sample conc > CRQL & > 5x blank value
Flag sample result with a "U";	Report CRQL & qualify "U"	No qualification is needed

NOTE: If gross blank contamination exists, all data in the associated samples should be qualified as unusable (R).

6.3 Are there field/rinse/equipment blanks associated with every sample?

☐ ☐ ☐

ACTION: Note in data assessment that there is no associated field/rinse/equipment blank. Exception: samples taken from a drinking water tap do not have associated field blanks.

## 7.0 Calibration and GC Performance

7.1 Are the following gas chromatograms and data systems printouts for both columns present for all samples, blanks?

a. peak resolution check

☐ ☐ ☐

b. performance evaluation standards  
(Continuing Calibration check)

☐ ☐ ☐



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YES    NO    N/A

c. aroclor 1016/1260	<input type="checkbox"/>	___	___
d. aroclors 1221, 1232, 1242, 1248, 1254	<input type="checkbox"/>	___	___
e. toxaphene	<input type="checkbox"/>	___	___
f. low level individual mixtures A & B	<input type="checkbox"/>	___	___
g. instrument blanks	<input type="checkbox"/>	___	___

ACTION: If no, take action specified in 3.1 above.

7.2 Are Forms VI HCA-1-2 present and complete for each column and each analytical sequence? ☐ \_\_\_ \_\_\_

Form VI HCA-1 must be completed for each level of the 3 point calibration. Therefore each sequence should have 3 copies of this form completed.

ACTION: If no, take action specified in 3.1 above.

7.3 Are there any transcription/calculation errors between raw data and Forms VI? \_\_\_ ☐ \_\_\_

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and document effect in data assessments.

7.4 The Relative Mean Deviation (RMD) for each peak must be < 0.5%. Absolute RT windows for major peaks are calculated as  $\pm 1.0\%$  of mean RT of standard. Are the RT of standards within RT windows established during initial calibration? ☐ \_\_\_ \_\_\_

ACTION: If no, all samples in the entire analytical sequence are potentially affected. If no peaks are found and the surrogates are visible, non-detects are valid. If peaks are present

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YES NO N/A

and cannot be identified through pattern recognition or using RT window, qualify all positive results and non-detects as unusable (R).

Aroclors and Toxaphene are identified primarily by pattern recognition, but RT's of 3 to 5 major peaks must be taken into consideration.

- 7.5 Were low level mixture of Individual Mixture A & B analyzed? ☐ ☐ ☐

NOTE: Low level mixture of single component pesticides are injected as part of the calibration sequence to establish the RT of individual pesticides since they are potential method interference.

- 7.6 Is Form IX HCA filled out correctly? ☐ ☐ ☐  
Elution order of compounds is different on each column. RT windows are  $\pm 1.5\%$  for 4 BHC and Heptachlor. The remaining compounds are  $\pm 1.0\%$ .

- 7.7 Are the linearity criteria for the initial analyses of Aroclors/Toxaphene within limits for both columns? ☐ ☐ ☐

NOTE: Linearity response is required for each of the 4 or 5 potential quantitation peaks selected during initial calibration, although only 3 peaks are needed for quantitation.

Form VI HCA-2 mean RT, mean CF, and %RSD values are based upon values reported on Form VI HCA-1.

- 7.8 Are the %RSD values for each continuing calibration (Performance Evaluation) <15%? (Form VI-2) ☐ ☐ ☐

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YES NO N/A

ACTION: If no, check linearity criteria below.  
If calibration fails criteria, qualify  
any associated positive results generated  
during the analytical sequence "J" and  
sample quantitation limits "UJ".

NOTE: Linearity criteria is based upon  
which method utilized by the lab.  
There are 3 methods allowed in SOW.  
Form VI HCA-2 is completed according  
to method chosen.

1. Mean CF can be used only if %RSD  
is <15%.
2. Single segment calibration line,  
regression coefficient  $r(1)$  should  
be >0.975 and Intercept (1) should  
be <0.20.
3. Two segment calibration line,  
regression coefficient (1 & 2)  
must be >0.975 and Intercept (1 & 2)  
must be <0.20 times the low point  
response.

Only 1 of 3 calibration methods can be used  
to quantitate samples in any single run.

7.9 Is the resolution between any two adjacent  
peaks in the Resolution Check Mixture > 60.0%  
for both columns?

☐ ☐ ☐

The method calls for a Resolution check to be  
analyzed and pass criteria although no Form  
is available in this protocol.

ACTION: If no, positive results for compounds  
that were not adequately resolved should  
be qualified "J". Use professional  
judgement to determine if non-detects  
which elute in areas affected by co-  
eluting peaks should be qualified "N" as

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YES    NO    N/A

presumptive evidence of presence or unusable (R).

- 7.10 Is Form VII HCA present and complete for each Performance Evaluation Standard analyzed during the analytical sequence for both columns?

[ ]    —    —

There is a specific timetable and standard that must be adhered to by lab. Check SOW ARO D-19, sec. 6.4.4.1 for time table.

ACTION: If no, take action as specified in 3.1 above.

- 7.11 Have all samples been injected within a 12 hr period beginning with the injection of an Instrument Blank?

[ ]    —    —

ACTION: If no, use professional judgement to determine the severity to the effect on data reliability.

- 7.12 Are there any transcription/calculation errors between raw data and Form VII HCA? (Form VII, %D).

—    [ ]    —

ACTION: If large errors exists, call lab for explanation/resubmittal, make any necessary corrections and document effect in data assessments under "Conclusions".

- 7.13 Do all standard retention times for each Continuing Calibration Standard fall within the windows established by the initial calibration sequence? ( $\pm 1.0\%$  of mean RT of initial calibration)

[ ]    —    —

ACTION: If no, beginning with the samples which followed the last in-control standard, check to see if the chromatograms contain peaks within an expanded window surrounding the expected retention times. If no peaks are found and the surrogates are visible,

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YES    NO    N/A

non-detects are valid. If peaks are present and cannot be identified through pattern recognition or using RT window, qualify all positive results and non-detects as unusable (R).

- 7.14 Are RPD values for all Evaluation standard compounds < 20.0%?

[ ]    \_\_\_    \_\_\_

ACTION: If the RPD is >20.0% for the compound being quantitated, qualify all associated positive results "J" and non-detects "UJ". The "associated samples" are those which followed the last in-control standard up to the next passing standard containing the analyte which failed the criteria. If the RPD is >90%, flag all non-detects for that analyte R (unusable).

8.0        Analytical Sequence Check (Form VIII HCA )

- 8.1 Is Form VIII HCA present and complete for each column and each period of analyses?

[ ]    \_\_\_    \_\_\_

ACTION: If no, take action specified in 3.1 above.

- 8.2 Was the proper analytical sequence followed for each initial calibration and subsequent analyses (see HC SOW Aro D-14 & D-18-19)?

[ ]    \_\_\_    \_\_\_

ACTION: If no, use professional judgement to determine the severity of the effect on the data and qualify it accordingly.

9.0        Cleanup Efficiency Verification (Form IX HCA)

- 9.1 Is Form II HCA present and complete for each lot of Diol Cartridges used? (Diol Cleanup is required for all Aroclor extracts.)

[ ]    \_\_\_    \_\_\_

ACTION: If no, take action specified in 3.1 above. If data suggests that Diol cleanup

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YES NO N/A

was not performed, make note in "Contract Problems/Non-Compliance".

9.2 Are all samples listed on the Aroclor Diol Cartridge Check Form? ☐ ☐ ☐

ACTION: If no, take action specified in 3.1 above.

9.3 Is the percent recovery (% REC) of the Aroclor 1254 used to check the efficiency of the cleanup procedures within QC limits ? 80-110% for Diol cartridge check? ☐ ☐ ☐

ACTION: If %REC of 1254 Aroclor is < 80%, qualify positive results "J" and quantitation limits "UJ" for these compounds.

If 1254 recovery is less than 10% all positive data should be qualified "J" non-detects should be qualified "R". Use professional judgement to qualify positive judgement to qualify positive results if recoveries are greater than the upper limit.

10.0 Pesticide/Aroclor Identification

10.1 Is Form X complete for every sample in which a PCB or Toxaphene was detected? ☐ ☐ ☐

ACTION: If no, take action specified in 3.1 above.

10.2 Are there any transcription/calculation errors between raw data and Forms ☐ ☐ ☐

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and note error under "Conclusions".

10.3 Are retention times (RT) of Aroclor peaks within the established RT windows for both

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analyses?

[ ] \_\_\_\_\_

NOTE: N flag identifies Aroclor or Toxaphene when 1 or more of the peaks used for quantitation are > 2 times the width of corresponding peaks in the highest concentration standard. It indicates an uncertainty in quantitation. Use professional judgement to qualify data.

11.1 Are the Aroclor Analysis Data Sheets (Form 1 Aroclor) present with required header information for each of the following:

a. samples? [ ]

b. Method Blanks? [ ]

c. Instrument Blanks? ☐

11.2 Are there any transcription/calculation errors in Form I results? Check at least two positive values. Were any errors found? [ ]

11.3 Are the CRQLs adjusted to reflect sample dilutions? [ ]

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YES NO N/A

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and document effect in data assessments.

ACTION: When a sample is analyzed at more than one dilution, the lowest CRQLs are used (unless a QC exceedance dictates the use of the higher CRQL data from the diluted sample analysis). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" value on the original Form I and substituting it with data from the analysis of diluted sample. Specify which Form I is to be used, then draw a red "X" across the entire page of all Form I's that should not be used, including any in the summary package.

ACTION: Quantitation limits affected by large, off-scale peaks should be qualified as unusable (R). If the interference is on-scale, the reviewer can provide an approximated quantitation limit (UJ) for each affected compound.

NOTE: If an acceptable chromatogram is achieved with a diluted cleaned sample extract, an additional analysis of 10 times the concentration of the dilution must be analyzed and reported with the sample data.

12.0 Chromatogram Quality

12.1 Were baselines stable? ☐ ☐ ☐

12.2 Were any electropositive displacement (negative peaks) or unusual peaks seen? ☐ ☐ ☐

NOTE: Aroclor and Toxaphene peaks, for standard and sample chromatograms must be visible (>25% of full scale) and well defined. If not the lab must be asked to resubmit



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YES NO N/A

expanded chromatograms. However the surrogate peaks should be always within the 100% range.

ACTION: Address comments under "System Performance" of data assessment

13.0 Field Duplicates

13.1 Were any field duplicates submitted for Aroclor/Toxaphene analysis?

☐ ☐ ☐

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative. However, if large differences exist, identification of field duplicates should be confirmed by contacting the sampler.